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TO: Interested Parties

FROM: Keith E. Kawaoka, D.Env., Program Manager
Hazard Evaluation and Emergency Response Office

SUBJECT: Pesticides in former agricultural lands and related areas - Updates on investigation and assessment (arsenic, technical chlordane, and dioxin test methodologies and action levels and field sampling strategies)

Attached for you information is a technical report that presents guidance on the assessment of pesticides in former agricultural lands and related areas. This report serves as an addendum to the Hazard Evaluation and Emergency Response (HEER) office document *Screening For Environmental Concerns at Sites With Contaminated Soil and Groundwater* (May 2005).

If you any question, place call Dr. Roger Brewer of my office at 1-808-586-4238 or contact him by email at roger.brewer@doh.hawaii.gov.

Attachment

Pesticides in Former Agricultural Lands and Related Areas
Updates on Investigation and Assessment

This technical report presents updated guidance on the investigation and assessment of residual pesticides in soils. The guidance focuses on the redevelopment of former agricultural land but is also applicable to golf courses, nurseries, military housing complexes and similar, large-scale projects involving soils that may have been treated with pesticides. Updates are provided for arsenic, chlordane and dioxin test methodologies and action levels. A basic review of multi-increment sampling strategies is also presented.

This technical report serves as an addendum to the Hawaiʻi Department of Health (HDOH), Hazard Evaluation and Emergency Response (HEER) document *Screening For Environmental Concerns at Sites With Contaminated Soil and Groundwater* (May 2005 and updates) and other related technical reports noted below. Information presented in these technical memoranda will be incorporated into the upcoming revision of the HEER office *Technical Guidance Manual.* Comment and suggestions are welcome. Please contact Dr. Roger Brewer of HDOH at 1-808-586-4328 or roger.brewer@doh.hawaii.gov for further information.

Pesticides of Potential Concern

"Pesticides" is a general term that includes any type of chemical mixture specifically formulated to kill "pests." Pesticides commonly used in Hawaiʻi include herbicides, fungicides and insecticides, the latter including termiticides and nematocides. HDOH recommends that sites where pesticides may have been regularly applied in the past be tested for residual contamination prior to redevelopment. The guidance is especially pertinent to large tracts of former agricultural land, golf courses, nurseries and military housing complexes that are being demolished and redeveloped with new homes.

In the case of former agricultural lands, contamination is likely to be heaviest in former pesticide mixing and staging areas, seed dipping areas and storage areas, although heavy contamination could occur in association with bagasse piles, settling ponds, former plantation camp areas, etc. Residual contamination in former fields has not been well documented, although HDOH is continuing to collect data for these areas. Conditions can vary dramatically from site to site.

Types of pesticides commonly used in Hawai'i include:

Pesticide Group & Related Contaminants	Standard USEPA Laboratory Method
Organochlorine pesticides	8081A
Organophosphorus pesticides	8041A
Chlorinated herbicides	8151A
Carbamates	8321A
Pentachlorophenol	8270
Fumigants	8260
Dioxins/furans	8280/8290
Heavy metals (primarily arsenic, lead & mercury)	various

The above list is not intended to be comprehensive, nor is it intended to represent a required list of target analytes that must be tested for in areas where pesticides were used in the past. Specific pesticides of concern should be based on a review of the historical use of the site with a focus on pesticides that may be persistent in soil above HDOH Environmental Action Levels (EALs, HDOH 2005 and updates). Soil and groundwater action levels for the majority of commonly used pesticides in Hawai‘i are included in this document. Contact the HEER office for pesticides not listed in the EAL document.

Fumigants are not likely to be persistent in shallow soils more than one year after use due to a propensity to volatilize into the atmosphere and degrade or be carried downward in leachate. Organochlorine pesticides are known to be very persistent in soils in Hawai‘i, as are arsenic and lead. Organophosphorus pesticides, chlorinated herbicides, carbamates and pentachlorophenol are more susceptible to biological and chemical breakdown over time and are more likely to be persistent in heavily contaminated, pesticide mixing areas than in fields. As of the date of the technical report, however, HDOH has not compiled adequate data to rule out the potential presence of these pesticides in former field areas above levels of concern. As discussed below, significant levels of dioxins and furans may also remain in soils even though the parent pesticide has degraded below levels of concern.

Summaries of historical pesticide use on agricultural lands are available from the Hawai‘i Department of Agriculture and other sources (e.g., Hanson 1959, 1962; HDOA 1969, 1977, 1989). The Clean Water Branch of HDOH provides a brief summary of pesticides in their NPDES guidance (HDOH 2004). A selection of pesticide-related documents can be downloaded from the HDOH EAL web page (refer to HDOH 2005).

A detailed review of pesticide use in Hawai‘i and a compilation of persistent contaminants that could accumulate in soil above levels of concern will be included in the upcoming revision to the HEER office Technical Guidance Manual (anticipated Fall 2007). Additional pesticides will be added to the current list of chemicals in the EAL lookup tables as needed.

Arsenic

HDOH recommends that the name of the soil series and a summary of the soil type be noted for samples tested for bioaccessible arsenic, including mention of the total iron and aluminum oxide content (NCRS 2007). HDOH also recommends that the fine-grained (<250µm) fraction of the soil sample that is to be tested for bioaccessible arsenic also be tested for total arsenic.

Guidance on the collection and interpretation of bioaccessible arsenic data is presented in the HDOH 2006 technical report *Soil Action Levels and Categories for Bioaccessible Arsenic* (HDOH 2006a). HDOH recommends that bioaccessible arsenic test be carried out when the total arsenic concentration in the soil exceeds 20 mg/kg (assumed upper limit for background arsenic in soil). Bioaccessible arsenic tests are used to estimate the fraction of total arsenic that could be stripped or “desorbed” from the soil following ingestion and thus made available for uptake. Arsenic that remains sorbed to the soil sample is considered to be unavailable for uptake and essentially “non-toxic.” The concentration of bioaccessible arsenic in a soil sample is calculated by dividing the mass of arsenic that moves into the batch test solution by the mass of the sample. Although not required as part of the bioaccessibility test, HDOH recommends that the concentration of total arsenic also be determined for the sample. This will help confirm the results of the test and provide insight on the range of arsenic bioaccessibility in the fine-grained fraction of contaminated soil.

The HEER Office has not developed generic bioaccessible factors for arsenic in soils in Hawaiʻi and currently recommends testing on a site-by-site basis. The use of bioaccessible arsenic tests has not been formally adopted by USEPA as a substitute for bioavailable arsenic testing (i.e. in-vivo or animal testing). In lieu of formal guidance, USEPA has recommended that HDOH provide additional information to demonstrate a preponderance of evidence that the arsenic is indeed tightly bound to the soil and has very limited availability for uptake in humans. These lines of evidence include:

- *In vivo* studies that indicate very low arsenic bioavailability in soils from heavily impacted areas in Keaʻau (Exponent 2005, Roberts et al., 2006);
- The correlation of *in vivo* study results with bioaccessible arsenic data collected at the same site (e.g., Cutler 2006);
- Correlation of decreasing arsenic bioavailability with increasing iron oxide concentration (Roberts et al., 2006);
- Average iron oxide concentration in soils used for agriculture in Hawaiʻi of 10-30%, well above typical soils on the US mainland (NRCS 2007);
- A lack of arsenic in groundwater underlying current and former sugar cane areas, indicating strong binding to soil and minimal leaching potential (HDOH 2006b);
- Laboratory testing at UH Manoa that demonstrated tropical soils (Andisols and Oxisols) with high levels of oxide and hydroxide mineral species have a natural ability to sequester arsenic, even over a wide pH range, making the arsenic less available to the soil solution - and therefore also estimated to be less bioaccessible through human ingestion and digestion (Cutler et al., 2006);
- Soil uptake factors for vegetables and fruits grown in arsenic-contaminated soils in the Keaʻau area are >2 orders of magnitude less than uptake factors published in scientific literature, supporting a conclusion that the arsenic is much more tightly bound to the soils than might otherwise be expected (HDOH, internal data);
- Laboratory batch test data that indicate arsenic sorption coefficients in soil greater than 500 (HDOH, internal data); and
- Use of a conservative, maximum-acceptable target risk to establish upper-bound action levels for bioaccessible arsenic in soil (HDOH 2006a).

HDOH recommends that a brief summary of the soil series associated with the subject site and sample point locations be provided with bioaccessible arsenic data (NRCVS 2007). The relationship between soil mineral characteristics and bioaccessibility is very complex and dependent on more than the metal oxide content of the soil. For example, the arsenic binding capacity of soils developed on coralline, coastal sediments is significantly less than soils developed over basalt, although these soils are rarely used for agriculture. Additional research is currently under way by the University of Hawaiʻi as well as other groups.

Technical Chlordane and Other Organochlorine Pesticides

HDOH recommends that soils potentially treated with termiticides be tested for *technical chlordane* rather than individual chlordane isomers and related compounds generally found in technical chlordane. The concentration of chlordane isomers, heptachlor and heptachlor epoxide do not need to be reported. Laboratories should be directed to test for technical chlordane using USEPA Method 8081A or an equivalent method (USEPA 1996). This must be specifically requested prior to submittal of the samples and noted on the Chain of Custody form. Laboratories should also be instructed to report any additional organochlorine pesticides that are not typically found in technical chlordane (e.g., DDT, dieldrin, endrin, etc.).

Technical chlordane is a mixture of chlordane (50-75%) and over 100 related compounds, including heptachlor and heptachlor epoxide (ATSDR 1994). Toxicity factors published by the USEPA are based on studies of technical chlordane, not individual chlordane isomers (USEPA 1997). These toxicity factors collectively take into account the full suite of compounds present in technical chlordane and are used to generate the USEPA Region IX Preliminary Remediation Goals (USEPA 2004) and HDOH Environmental Action Levels (HDOH 2005) for direct-exposure concerns. Since the quantification of technical chlordane includes chlordane, heptachlor and heptachlor epoxide, these individual compounds do not need to be reported in the analysis or evaluated separately in an Environmental Hazard ("risk") Assessment unless otherwise directed by HDOH. Doing so will cause the health risk posed by these compounds to be double counted, since it will already be included in the assessment of technical chlordane.

Tier 1 Environmental Action Levels (EALs) for technical chlordane are presented in the HDOH document *Screening For Environmental Concerns at Sites With Contaminated Soil and Groundwater* (HDOH 2005). Tier 1, direct-exposure action levels technical chlordane were generated using a target excess cancer risk of 10^{-6} and a noncancer hazard quotient of 1.0 (refer to Appendix 1, Tables I-1 through I-3). However, use of a target, cumulative risk of 10^{-5} is generally acceptable for evaluation of multiple contaminants under a Tier 2 or Tier 3 assessment. Since technical chlordane is actually a mixture of numerous chemicals, it is more appropriate to screen site data using Tier 2 direct-exposure action levels based on a cumulative target cancer risk of 10^{-5}. Correlative action levels for residential and commercial/industrial exposure are 16 gm/kg and 65 mg/kg, respectively. After taking into account action levels for leaching concerns, the following action levels are generated:

Tier 2 Action Levels for soil with technical chlordane only

Exposure Scenario	**Direct Exposure (mg/kg)**	**[1]Leaching (mg/kg)**	**[2]Final Tier 2 Action Level (mg/kg)**
Residential	16	15	15
Commercial/Industrial	65	15	15

1. HDOH EAL guidance document, Appendix 1, Table E-1.
2. Lowest of direct-exposure and leaching soil action level

These action levels will replace the Tier 1 action levels for technical chlordane currently presented in the HDOH EAL document (HDOH 2005). On a site-specific basis, HDOH may require calculation of cumulative cancer and noncancer health risks if contaminants not related to technical chlordane are identified in the soil above HDOH Tier 1 action levels for direct-exposure concerns. This may be necessary to assure that a cumulative cancer risk of 10^{-5} and a noncancer Hazard Index of 1.0 are not significantly exceeded. Leaching concerns posed by the additional contaminants must also be evaluated.

Refer to the HDOH technical report *Use of laboratory batch tests to evaluate potential leaching of contaminants from soil* for guidance on the site-specific evaluation of potential leaching concerns and the development of alternative action levels (HDOH 2007). Technical chlordane has a very low mobility in soil and the soil leaching action level is considered to be highly conservative. If the batch tests indicate that the technical chlordane does not pose a threat to groundwater then the direct-exposure action levels (or estimated cumulative health risks) can be used to guide final remedial actions.

Soil that meets the Tier 2, commercial/industrial direct-exposure action level for technical chlordane (i.e., up to 65 mg/kg) can be used as interim (daily) cover in landfills, provided that the soil passes a TCLP leaching test and given the concurrence of the landfill operator. A maximum concentration of 65 mg/kg technical chlordane is recommended, unless otherwise approved by HDOH. Soil used for longer-term, intermediate cover must meet more stringent action levels (e.g., residential). Contact the HDOH Solid and Hazardous Waste Branch for additional information regarding the use or disposal of soil at landfills.

Dioxin

HDOH concurs with the use of bioassay kits to help reduce the time and expense related to investigation of dioxin concentrations in soil. Dioxins are included in Table 1 as potential contaminants of concern due to their presence in pesticides (NTP 2005), especially pentachlorophenol (PCP), 2,4,5-trichlorophenoxyacetic acid (2,4,5-T) and 2,4,5-trichlorophenoxypropionic acid (2,4,5-TP or Silvex). These chemicals were used as herbicides on agricultural lands in Hawai‘i. Dioxins can also be created when organic material is burned in the presence of chlorine, including the burning of sugar cane fields where organochlorine pesticides and other chlorine containing media are present.

Quantification of dioxins in soil is for use in human health risk assessments is carried out using GC/MS laboratory methods (USEPA Methods 8280 and 8290). Risk to human health is estimated in terms of toxicity equivalents of individual dioxin and furan congeners or “TEQ dioxins” (WHO 2005). Soil action levels for TEQ dioxins are discussed in the HDOH technical report *Proposed dioxin action levels for East Kapolei Brownfield Site* (HDOH 2006c).

Laboratory GC/MS tests can be expensive and time consuming, with analytical costs typically ranging between $750 and $1,000 per sample. Bioassay methods offer a cheaper and faster approach to screen for dioxins in soils. Bioassay methods currently available include CALUX (Dennison et al. 1999, USEPA 2005a) and Cape Technology’s DF1 kit (USEPA 2005b). Bioassay data are reported directly in terms of TEQ units. In order to evaluate the accuracy and precision of bioassay kits for soils in Hawai‘i, HDOH collected 25 soil samples from a former sugar cane field in west O‘ahu and tested the samples for TEQ dioxins using both High Resolution GC/MS and CALUX. A summary of the results of the study is presented in Figure 1. As can be noted in the figure, CALUX consistently over predicted TEQ dioxin concentrations in the soil in comparison to the GC/MS analysis. While the correlation of the CALUX test with the GC/MS data is somewhat low, the conservative nature of the CALUX test supports its use as screening tool to estimate maximum levels of TEQ dioxins in soil.

For sites where a bioassay method is used for dioxin analysis, HDOH recommends that dioxin levels be confirmed on 10% of the samples using GC/MS (or two samples, whichever is greater). The GC/MS analyses should be conducted on samples with the highest-reported, bioassay TEQ dioxins results. Additional analysis of samples using GC/MS methods may be necessary for sites where CALUX tests indicate TEQ dioxins in soil over the HDOH upper action level of 390 ng/kg.

Refer to the HDOH technical report *Proposed dioxin action levels for East Kapolei Brownfield Site* for additional guidance on soil action levels (HDOH 2006c).

Multi-Increment and Decision Unit Investigation Strategies
HDOH strongly encourages the use of *multi-increment* and *decision unit* strategies (Ramsey and Hewitt 2005) to enhance sample representativeness in the investigation of contaminated soil. Multi-increment samples significantly increase the accuracy of representative contaminant concentrations, in comparison to traditional, discrete samples (Jenkins et al. 2005). Establishing decision units early in the investigation helps integrate the field investigation with an assessment of potential environmental concerns, referred to as an "Environmental Hazard ("risk") Assessment" (HDOH 2005).

Detailed guidance on multi-increment and decision-unit investigation strategies will be included in the upcoming revision to the HEER Office Technical Guidance Manual (anticipated late 2007). In the interim, a brief summary of these approaches is provided below. Example work plans and site investigation reports can be reviewed at the HEER office and will be posted to the HDOH EAL web page (refer to HDOH 2005). Reviewers of these reports should be aware that the projects were carried out during ongoing refinement of approaches for the investigation of large areas and the sampling strategies presented may not be directly transferable to new sites without modification and consultation with HDOH.

<u>Multi-Increment Samples</u>
Multi-increment samples improve the reliability of sample data by reducing the variability of the data as compared to conventional discrete sampling strategies. Thirty to fifty small *increments* of soil (typically 10 to 50 grams per increment) are collected from each specific *decision unit* of interest (see below). The increments are collected in a stratified-random manner (e.g., by walking up and down adjacent rows) and physically combined into one sample. The combined sample is analyzed to obtain a representative contaminant concentration for the entire decision unit. Multi-increment sampling data typically have low variability and high reproducibility, which results in a high level of confidence for decision-making. Three multi-increment samples, referred to as field replicate samples or *triplicates*, should be collected in 10% of the decision units being tested (minimum one set of triplicate samples per site). Data for the samples can be statistically compared in order to evaluate the precision of the field sampling methodology.

Multi-increment samples generally weigh between 500 and 2,000 grams. The laboratory dries the sample, sieves it to <2mm particle size (can also be done in the field) and collects a subsample for analysis. To obtain a representative sub-sample, the field sample must be processed so that the entire "population" of soil particles is accessible for collection. Sub-sampling can be accomplished with a sectoral splitter or by collecting a multi-increment sample using the same approach as used to collect the field sample but with smaller tools and increment masses (USEPA 2003). A larger mass than typically called for in the published USEPA laboratory method is recommended for in order to reduce lab fundamental error due to the range of particle sizes being tested (e.g. 10 gram versus 1 gram sample for total arsenic analyses, based on a maximum particle size of 2 mm). [Note that a mass of 1 gram is considered acceptable for samples that have been sieved to <250µm, as is required for bioaccessible arsenic analysis.]

Multi-increment samples can be collected for both nonvolatile and volatile contaminant analyses. Sample collection for volatile contaminants requires that increments be placed in an extraction solution in the field (ADEC 2007). Issues related to field extraction solutions, methanol transportation in the field, appropriate sample containers, elevated laboratory method reporting limits, etc., still need to be worked out, however, and this approach has not yet been widely used in Hawai‘i. Additional guidance will be provided in the upcoming revision of the HEER office *Technical Guidance Manual* (anticipated late 2007). Consultants who would like to use the approach in the meantime should provide sampling and analysis work plans to the HEER office for review and ensure close coordination with the receiving laboratory.

<u>Decision Units</u>
Multi-increment samples should be collected in carefully selected *decision units*. A decision unit is an area where a decision is to be made regarding the extent and magnitude of contaminants with respect to the environmental concerns posed by the contaminants. (Strictly speaking, a decision unit is really a volume rather than area of soil, since the thickness of the decision unit is often a key factor.) These concerns include direct exposure to the soil, intrusion of vapors into overlying buildings, leaching and contamination of groundwater, toxicity to terrestrial flora and fauna and gross contamination (odors, explosive hazards, etc.; HDOH 2005). A decision unit can be an identified spill area or “hot spot,” a residential yard, a playground or schoolyard, a garden, a commercial/industrial property or other specific area of interest. The size and shape of a decision unit is primarily controlled by the environmental concerns posed by the contaminants present and the intended use of the site.

An investigation of individual spill areas is generally necessary to assess leaching, vapor intrusion and gross contamination concerns at sites contaminated with highly mobile or volatile contaminants. This can include releases from pipelines or tanks or heavily contaminated portions of pesticide mixing areas in former agricultural lands. Each spill area represents a single decision unit. Discrete samples, or more preferably multi-increment samples collected over small areas, can be useful for delineation of spill area boundaries. The spill areas themselves should be sampled using multi-increment samples when feasible, however. Non-volatile contaminants in spill areas can be readily sampled using multi-increment sampling methods. Volatile contaminants could also be investigated with multi-increment sampling in these areas, although guidance on field methods has yet to be worked out in detail (see above).

Decision units that encompass an entire residential or commercial lot are appropriate for assessment of direct-exposure concerns. This is typically the driving environmental concern for the investigation of former agricultural field areas. Each residential lot represents a separate decision unit. Testing every lot may not be feasible or necessary for projects over 10 to 25 acres in size, depending on the size of the individual lots (default is 5,000 ft^2). For moderate-size sites, it may be feasible to combine multiple lots into larger “composite” decision units (typically up to five lots) and collect a single multi-increment sample from within each unit. The maximum concentration of a contaminant in any given lot is equal to the concentration reported for the composited decision unit times the number of lots included (i.e., assumes all of the contamination is on one lot). The variance of contaminant concentrations between composited decision units may also be useful to estimate worst-case contaminant concentrations on individual lots.

For very large redevelopment projects (e.g., >100 acres), testing each individual lot and even combining lots into larger decision units may not be practical. As an alternative, HDOH recommends that multi-increment samples be collected from a statistically defendable number of 5,000ft^2 decision units randomly located across the site. Each decision unit represents a hypothetical, residential lot. The data from these decision units can be statistically evaluated to predict maximum contaminant concentrations on any given lot within the site. Although not every lots is tested, this approach ensures that data are at least available for nearby, presumably comparable lots.

A minimum of 59 decision unit is required to obtain the HDOH-desired, 95% confidence level that residual levels of pesticides on untested lots do not exceed the maximum concentration identified on the lots tested (USEPA 1989). Past crop types, topography, soil type, planned future use and related factors should be considered in the selection of decision unit locations in order to ensure that a representative sampling of the site is carried out. Areas suspected of potentially higher levels of contamination should be investigated separately and not included in the 59 decision units selected to characterize the primary field area (e.g., former pesticide mixing areas, storage areas, plantation camps, rail lines, etc.) This approach has been used at several large-scale redevelopment sites in Hawaiʻi and will be discussed in the upcoming revision of the HEER office Technical Guidance Manual.

Initial Screening of Agricultural Lands

It is often desirable to carry out a screening level investigation of former agricultural land prior to committing funds for a full-scale, detailed investigation, as described above. Although not adequate for HDOH to make final regulatory determinations, this step provides important information that can be used to prepare a more detailed work plan. Defensible methods to screen large areas of land are still being developed. A combined multi-increment/decision unit approach is preferred over the collection of a limited number of discrete or composite samples based on the total acreage of the site. Two example approaches are described below.

A relatively quick and sensible approach is to divide the site into neighborhood-size decision units rather decision units based on the size of hypothetical, individual lots. An area of ten acres is a reasonable starting point for a "neighborhood." A minimum of 15 decision units per site is preferable. This helps to ensure coverage of large-scale heterogeneities across the site and, if needed, is usually adequate for use in basic statistical analyses. The size and shape of individual decision units can vary and should be determined with respect to soil type, topography, past crop use, proposed redevelopment, etc., as discussed above. A multi-increment sample should be collected from each decision unit, with triplicates collected in ten-percent of the decision units (minimum two). Each sample should be tested for the full suite of pesticides that may have been used at the site in the past, including related contaminants like arsenic and dioxins. Again, areas suspected of higher levels of contamination should be investigated separately.

An alternative approach for sites where access is an issue is to collect multi-increment samples in 18 (vs 59), 5,000ft^2 decision units randomly located across the site, each representing a hypothetical, residential lot. This allows an estimation of maximum contaminant levels on any given lot to a 60% confidence level (USEPA 1989). The samples should be tested as described above. The collection of multi-increment samples in specified decision units is preferred to the collection of randomly located, discrete samples in large field areas.

As is often the case, developing the most appropriate investigation for a given site involves a balance of short-term time versus long-term uncertainty and liability. A neighborhood-based, screening level investigation is recommended where feasible. Testing entire neighborhoods will provide some level of comfort to future residents whose lots were not tested during the full-scale investigation. Perhaps most importantly, however, this approach requires a thorough walkthrough of the entire site. This will assist in the identification of areas suspected of elevated contamination, including previously unknown dumping sites, waste pits, former plantation camp areas, storage areas, etc. When walking and testing the entire site is not feasible, testing a limited number of lot-size decision units is recommended. One advantage of this approach is that the decision unit data can potentially be included in the full-scale investigation of the property (e.g., 18 of the 59 total decision units), saving on follow up investigation time costs.

Sample data from the screening level investigation should be used to initially assess residual pesticide levels in the fields and to prepare a more focused list of target pesticides of concern for the detailed investigation. Pesticides that are not detected during the initial screening investigation can generally be eliminated, although this should be discussed with HDOH. Eliminating specific pesticides from the list of target contaminants will require approval if the site is being formally overseen by HDOH.

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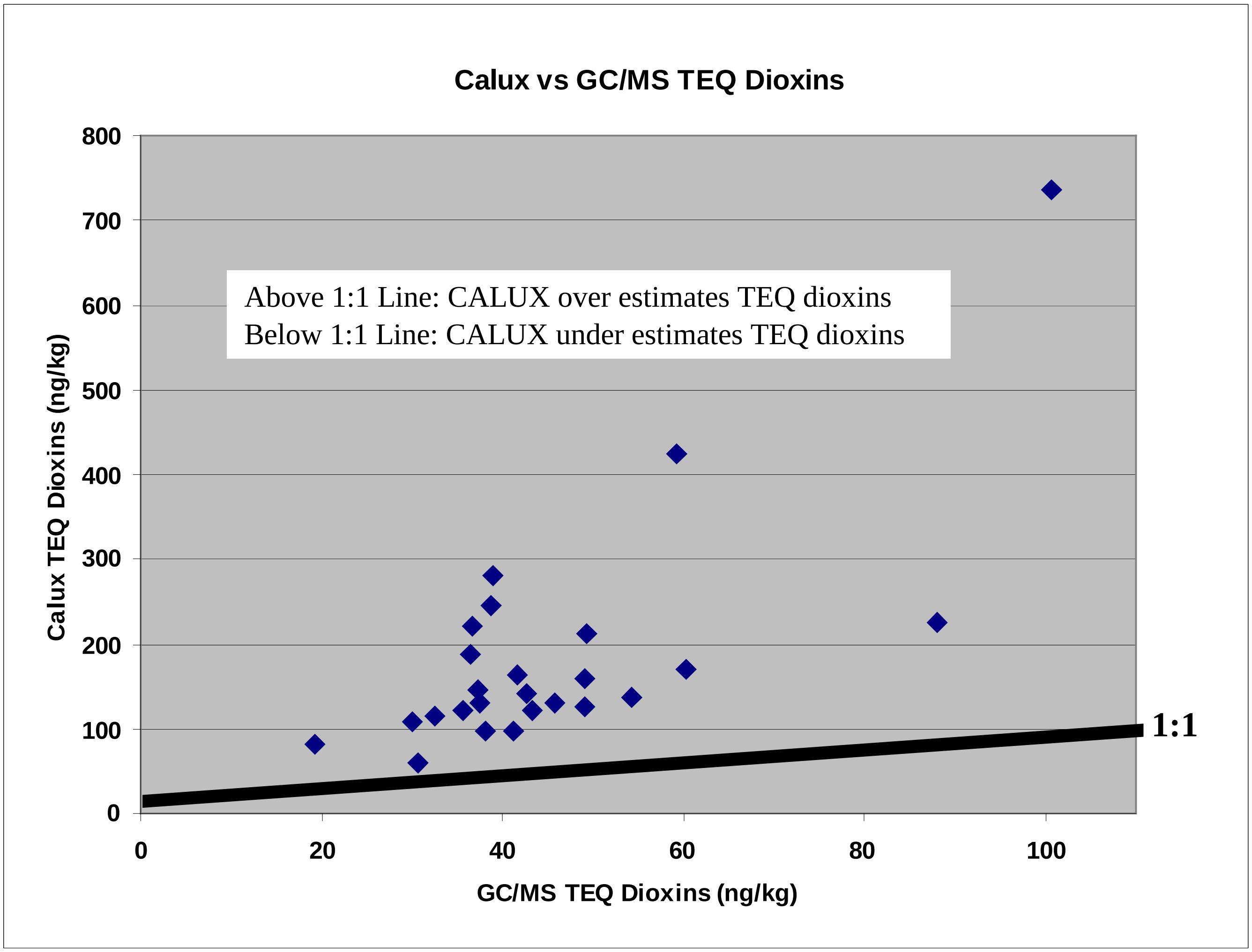

Figure 1. Results of East Kapolei study conducted on 25 samples of soil from a former sugar cane field. CALUX consistently over estimated TEQ dioxins in comparison to High Resolution GC/MS data for split samples.